

OFFICE OF NAVAL RESEARCH

Contract N00014-91-J-1078

R&T Code 4132046

Technical Report No. 35

FERROELECTRIC NYLON-7, -11 ULTRASONIC TRANSDUCER PERFORMANCE

by L.F. Brown, J.L. Mason , D. Khataniar, J.I. Scheinbeim and B.A. Newman

Prepared for Publication
in

Proceedings of the 1995 IEEE International Ultrasonics Symposium

Department of Electrical Engineering
South Dakota State University
201 Harding Hall
Brookings, SD 57007

Polymer Electroprocessing Laboratory
Department of Chemical and Biochemical Engineering
College of Engineering
Rutgers University
Piscataway, NJ 08855-0909

April 1996

Reproduction in whole or in part is permitted for any purpose of the United States Government.

This document has been approved for public release and sale; its distribution is unlimited.

DTIC QUALITY INSPECTED 3

19960508 190

Ferroelectric Nylon-7, -11 Ultrasonic Transducer Performance

By

L.F. Brown¹, J.L. Mason¹, D. Khataniar¹, J.I. Scheinbeim², and B.A. Newman²

¹Department of Electrical Engineering

South Dakota State University

201 Harding Hall

Brookings, SD 57007

Ph.: (605) 688-4526, Fax: (605) 688-5878

²Polymer Electroporessing Laboratory

Department of Materials Science and Engineering

Rutgers University

P.O. Box 909

Piscataway, NJ 08855-0909

Ph.: (908) 445-3669, Fax: (908) 445-0654

Presented at

The 1995 IEEE International Ultrasonics Symposium

Seattle, WA USA

November 7-10, 1995

Ferroelectric Nylon-7, -11 Ultrasonic Transducer Performance

L.F. Brown¹, J.L. Mason¹, D. Khataniar¹, J.I. Scheinbeim², and B.A. Newman²

¹South Dakota State University, Brookings, SD 57007

²Rutgers University, Piscataway, NJ 08855-0909

Abstract--Measurements of the high frequency electrical and acoustic properties of ferroelectric nylon 7 and 11 are reported for frequency and temperature ranges of interest for ultrasonic transducer applications in medicine and nondestructive testing. The dielectric properties of the samples were measured over a broadband frequency range (typically 1-100 MHz), including the fundamental half-wave resonance frequency for the sample, and the properties of interest for ultrasound transducer design were determined using a curve-fitting resonance technique. These properties were then used in a computer simulation program to assess the acoustic performance of nylon-based ultrasonic transducers and compare them with PVDF and P(VDF-TrFE) copolymer designs. Comparisons were carried out on the pulse-echo acoustic performance including pulse width, sensitivity, insertion loss, and bandwidth. Finally, a nylon ultrasound transducer was fabricated for pulse-echo acoustic performance measurements and comparison with PVDF. Tests verified the broadband acoustic performance of nylon 7.

I. INTRODUCTION

The ferroelectric (odd-numbered) nylons have generated recent interest for high temperature transducer applications which possibly cannot be addressed with other piezo/pyro/ferroelectric materials. With bulk material properties similar to PVDF and other polymers, the nylons may offer superior performance for high temperature applications which are beyond the safe operating temperature of PVDF and its copolymers. The purpose of this work was to study the basic dielectric and electromechanical properties of the nylons to assess their potential for high frequency transducer applications. Of particular interest was their high frequency thickness-mode properties which are important to the operation of resonance-mode ultrasonic transducers. This work was carried out on both nylon 7 and nylon 11 materials.

The odd-numbered nylons are known to possess a polar crystalline form which can be rendered ferroelectric through electroprocessing [1]. Extensive studies have been conducted on the low frequency dielectric and piezo/ferroelectric properties of nylon 7 and nylon 11 [2], but only limited work has been reported on their high frequency properties of interest [3]. The higher dipole

density of the lower ordered nylons, such as nylon 5, and nylon 7, results in predictably higher remanent polarization which has been shown to be competitive with PVDF and PVDF-based copolymers.

This paper describes the preparation of nylon 7 and nylon 11 samples, results of their dielectric and electromechanical characterization, and the results of computer simulations of their ultrasonic transducer performance. Test results from the first nylon ultrasonic transducer are also reported.

II. SAMPLE PREPARATION

Nylon 7 and nylon 11 film samples were produced at Rutgers University using the same melt quenched film technique reported elsewhere [1-3]. Thin gold electrodes, with 10 mm by 10 mm overlap, were evaporated onto opposing surfaces of the films to facilitate electro-processing and characterization. The samples were molecularly oriented by uniaxially drawing them to a ratio of approximately 2.8:1, which leads to higher attainable remanent polarization (i.e., higher crystallinity is imparted). The typical remanent polarization of the oriented nylon 11 samples measured 52 mC/m² while that of the oriented nylon 7 samples measured 80 mC/m². Polarization was carried out using a maximum electric field of 250 MV/m as described elsewhere.

Following polarization, the thickness of each sample was mapped and test specimens of smaller size were selected for maximum thickness uniformity and highest surface quality. The area of each test specimen was selected such that the magnitude of its impedance was in the range of 50-250 Ω at the fundamental half-wave thickness mode resonance frequency of the sample. This assured accurate measurement of the broadband dielectric properties near resonance.

III. SAMPLE CHARACTERIZATION

Test specimens were connected to a test fixture and remotely located in a Thermotron environmental chamber. The free-air capacitance and dielectric loss tangent ($\tan\delta_c$) were measured with a Hewlett-Packard 4195A Impedance Analyzer over a broad range of frequencies including the fundamental thickness mode resonance of the specimen.

Resonance was easily detected by the sharp changes in dielectric properties which occur when the sample thickness is 1/2 wavelength. The measurements were then analyzed using a resonance technique method [4, 5] whereby the parameters of a one-dimensional thickness-mode Mason's model were varied until the features of the model's input dielectric properties matched those of the sample within a specified tolerance.

Figure 1 shows the results of the nylon 11 measurements and those for nylon 7 are shown in Figure 2. Both samples were characterized at 20°C temperature increments in the range of 20-160°C. With a higher remanent polarization in the nylon 7 sample, a greater difference was expected in the dielectric and electromechanical properties between these two materials.

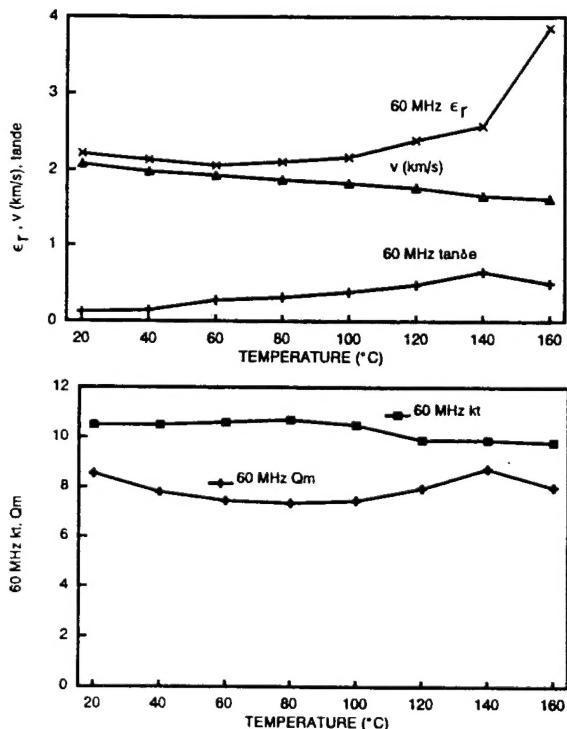


Fig. 1. Dielectric and electromechanical measurement results for oriented nylon 11 sample.

The results show a very low Q_m , and a k_t which is lower than typical PVDF, but is stable to 160°C--well beyond the range of commercially available PVDF and most of its copolymers. A direct comparison with PVDF and P(VDF-TrFE) copolymer properties is shown in Table 1.

Of greatest concern for low frequency medical applications (i.e., 1-10 MHz) is the low dielectric constant and high dielectric loss tangent of the nylons. The low mechanical Q and very low acoustic impedance of the nylons should offer greater bandwidth than PVDF and P(VDF-TrFE) for an ultrasonic transducer operating in

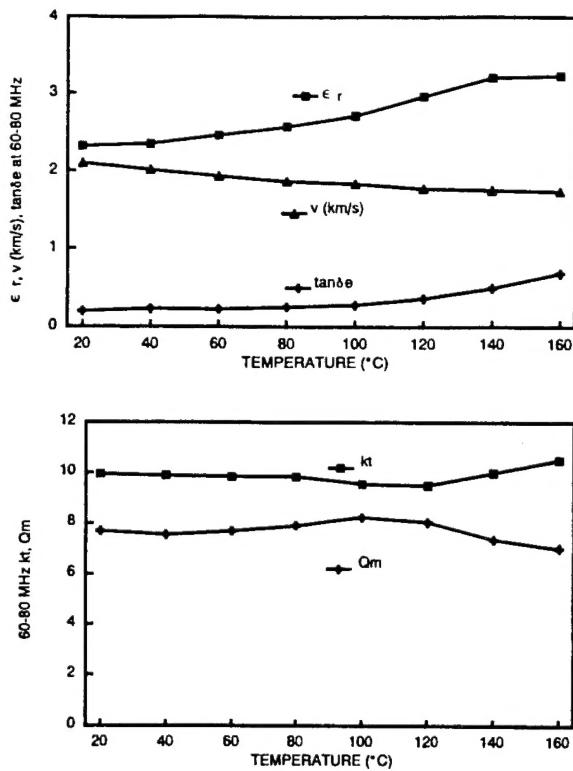


Fig. 2. Dielectric and electromechanical measurement results for oriented nylon 7 sample.

water or tissue. However, the advantage is likely overcome by the nylons' lower electromechanical coupling and higher internal dielectric and mechanical losses. Whether or not the nylons can offer definite advantages over PVDF or P(VDF-TrFE) as an ultrasonic transducer would likely depend on the physical requirements of the transducer and operating temperature. The measured nylon properties were used in numerous computer simulations in an attempt to assess their expected performance in ultrasonic transducers.

IV. COMPUTER SIMULATIONS

The results of the materials measurements summarized in Table 1 were utilized in Mason's model to simulate the performance of 5 MHz polymer ultrasonic transducers. Of particular interest was a relative comparison between the nylons and PVDF materials for pulse-echo operation. The ideal impulse response of each design was computed for pulse-echo operation, assuming a 50Ω transmitter/receiver system. The typical 20-100°C properties of each material were used in the simulations. In order to assure the optimum operation of each design, the diameter for each transducer model was selected so that the corresponding capacitive reactance of the polymer material was 50Ω at resonance. Since the high frequency dielectric constants differ for all of the polymer materials,

Table 1. Comparison of oriented nylon 7 and nylon 11 with typical high-frequency properties of PVDF and P(VDF-TrFE) at 20-100°C.

Parameter	PVDF	P(VDF-TrFE)	Oriented Nylon-7	Oriented Nylon-11
ϵ_r	5.0	4.0	2.5	2.2
$\tan\delta_c$	0.25	0.12	0.22	0.25
k_t	0.15	0.30	0.10	0.11
Q_m	13	25	8	8
v (m/s)	2200	2400	2000	2000
Z (MRayl)	3.96	4.27	2.30	2.30

so did the diameter for their corresponding ultrasonic transducer model design. Table 2 summarizes the film thicknesses (selected for $\lambda/2$ resonance at approximately 5 MHz) and transducer diameter used for each of the materials.

Table 2. Polymer transducer designs.

Transducer Material	Transducer Diameter (in.)	Film Thickness (μm)
P(VDF-TrFE)	2.92	240
PVDF	2.50	220
Nylon-11	3.59	200
Nylon-7	3.37	200

The Mason's model computer software was used to assess the ideal impulse response of each transducer operating in the pulse-echo mode. The frequency and time domain results are shown in Figure 3 and are quantitatively summarized in Table 3. As expected, the P(VDF-TrFE) copolymer design yielded superior ultrasonic performance to PVDF and the nylon materials. The insertion loss for the nylon transducers is poorer by approximately the square of the ratio of k_t for the P(VDF-TrFE) copolymer to that of the nylons (≈ 9 V/V or 19 dB). One comparison of interest is the pulse-width response of the polymer transducers. The nylon transducers, because of their closer acoustic impedance match to water, yielded slightly better responses.

These computer simulations show that the nylon materials offer ultrasonic transducer performance which can be close to that of PVDF, but well below that available with P(VDF-TrFE) copolymer designs in the 20-100°C temperature range. Their lower dielectric constant requires that nylon transducers have considerably larger area than those designed with PVDF and P(VDF-TrFE) copolymer.

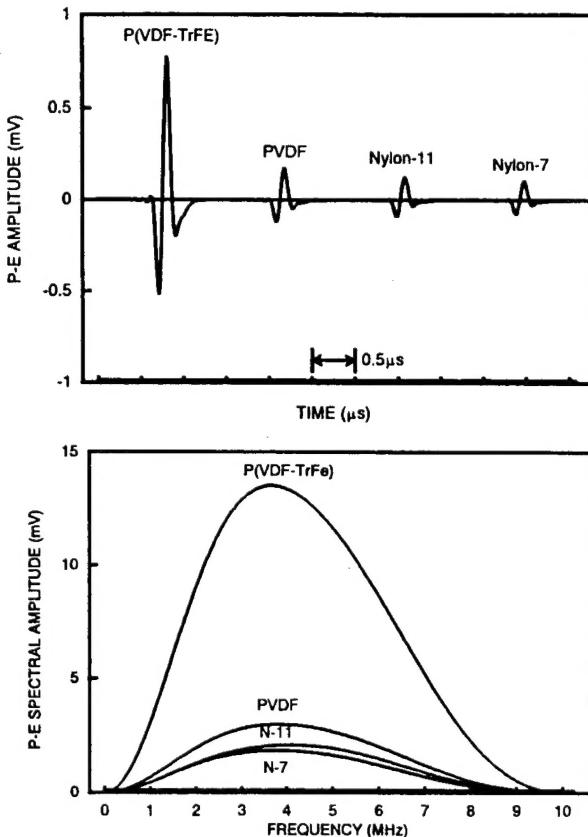


Fig. 3. Computer simulation results.

V. NYLON TRANSDUCER TESTS

To observe the actual pulse-echo ultrasonic performance of a nylon transducer, a simple nylon 7 transducer was constructed for laboratory measurements. A gold metallized nylon sample was selected which had an active area of approximately 1 cm^2 , and thickness of 38

Table 3. Summary of computer simulation results.

Material	I.L. _{min} (dB)	F _c (MHz)	-6 dB Bandwidth	-20 dB Pulse-width
P(VDF-TrFE)	37.4	4.1	120%	0.354 μ s, 1.5 λ
PVDF	50.5	4.2	121%	0.330 μ s, 1.4 λ
Nylon-11	53.4	4.2	121%	0.317 μ s, 1.3 λ
Nylon-7	54.8	4.2	120%	0.317 μ s, 1.3 λ

μ m. The test transducer featured an acrylic backing to produce maximum bandwidth and $\lambda/2$ resonance while oil coupled to a 3/16"-thick acrylic test plate. The transducer was connected to a Panametrics 5052PR Pulser/Receiver (Energy=3, Damping=6, Attenuation=10 dB, Gain=20 dB), and Tektronics 2440A Digital Storage Oscilloscope (50 Ω input impedance). The first acoustic back wall reflection in the acrylic test block was captured on the oscilloscope and transferred to a pc via IEEE-488 bus. Figure 4 shows that pulse-echo waveform and its corresponding frequency spectrum for the nylon 7 transducer. For these test conditions the pulse-echo waveform revealed a center frequency of 8.8 MHz and -6 dB fractional bandwidth of 144%. The results show extremely broadband acoustic performance, as expected since the acoustic impedance of the nylon (2.3 MRayl) is very close to that of the Plexiglas backing and test plate (3.1 MRayl).

VI. CONCLUSIONS

Ferroelectric nylon 7 and nylon 11 films have been characterized for their high frequency dielectric and electromechanical properties of importance for ultrasonic transducer performance, over the temperature range of 20-160°C. The electromechanical coupling constant k_t and mechanical Q were lower than that typically seen for PVDF and P(VDF-TrFE) copolymers, resulting in poorer ultrasonic transducer performance despite the nylon's lower acoustic impedance. The nylon materials also have a much lower permittivity, requiring much larger active transducer areas for efficient transducer designs. Computer simulations, and actual ultrasonic measurements on the first nylon ultrasound transducer, both verified that the ferroelectric nylon materials can achieve ultrasonic performance close to that of PVDF and can operate at higher temperatures. Better performance is anticipated for nylon 5, based on its higher dipole density and remanent polarization measurements. This work further confirms the potential of ferroelectric nylon materials to be used for high frequency sensor applications.

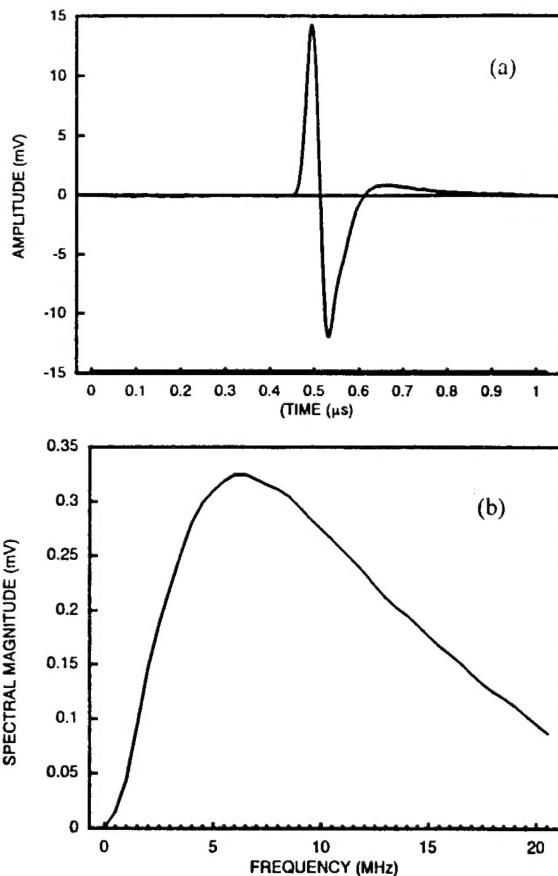


Fig. 4. Actual pulse-echo measurements for the oriented nylon 7 transducer: (a) time domain back wall reflection, (b) corresponding frequency spectrum.

REFERENCES

- [1] J. Scheinbeim, J. Lee, and B. Newman, "Ferroelectric Polarization Mechanisms in Nylon-11", *Macromolecules*, Vol. 25, 1992, pp. 3729- 33.
- [2] J.I. Scheinbeim and B.A. Newman, "Electric Field-Induced Changes in Odd-Numbered Nylons", *Transient Polymer Science*, Vol. 1, No. 12, Dec., 1993, pp. 394-400.
- [3] L.F. Brown, J.I. Scheinbeim, and B.A. Newman, "High Frequency Dielectric and Electromechanical Properties of Ferroelectric Nylon 11", Proc. 9th ISAF, IEEE Cat. No. 94CH3416-5, pp. 337-340. 1995.
- [4] H. Ohigashi, "Electromechanical Properties of Polarized Polyvinylidene Fluoride Films as Studied by the Piezoelectric Resonance Method", *Journal of Applied Physics*, Vol. 47, No. 3, March 1976, pp. 949-955.
- [5] L.F. Brown, "Disposable PVDF Ultrasonic Transducers for Nondestructive Testing Applications", accepted for pub. in *IEEE Trans. Ultras., Ferroelec. Freq. Contr.*